

WHAT IS CLAIMED AS NEW AND IS DESIRED TO BE SECURED BY LETTERS
PATENT OF THE UNITED STATES IS:

1. A process for preparing stable and high-solids aqueous dispersions of blocked isocyanates, containing auxiliary solvent,
5 which comprises:

(1) preparing a solution of a polyisocyanate mixture of from 20-70% by weight of a blocked, hydrophilically modified polyisocyanate (A) and from 30-80% by weight of a blocked, hydrophobic polyisocyanate (B) in an auxiliary solvent (G); and

10 (2) adding to this solution, with intensive mixing, an amount of water which is such as to give a two-phase system comprising a disperse polyisocyanate phase and a continuous, aqueous phase containing auxiliary solvent.

15 2. The process as claimed in Claim 1, wherein the blocked, hydrophilically modified polyisocyanate (A) is a blocked, ionic hydrophilically modified polyisocyanate which forms as a result of complete or partial neutralization of a polyisocyanate having a potentially hydrophilic group.

20 3. The process as claimed in Claim 2, wherein the potentially hydrophilic group is a carboxyl group and the neutralizing agent is ammonia or an amine.

25 4. The process as claimed in Claim 1, wherein the mixture of the polyisocyanates (A) and (B) is produced by only partially hydrophilicizing an amount of hydrophobic polyisocyanate (C) corresponding to this mixture by reacting it with an amount of hydrophilicizing agent (E) which is such that the proportions of resultant, hydrophilically modified polyisocyanate (D) and of

RECEIVED
JULY 22 1980
15
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
95

5 remaining hydrophobic polyisocyanate (C) correspond to the desired ratio of blocked hydrophilically modified polyisocyanate (A) to blocked hydrophobic polyisocyanate (B) for the finished dispersion, by converting any potentially hydrophilic groups by neutralization into ionic hydrophilic groups and by blocking the unreacted isocyanate groups of the hydrophilicizing mixture.

5. The process as claimed in Claim 1, wherein dispersions, which are virtually free from auxiliary solvent are prepared by:

(3) adding a first portion of the total amount of water used in the preparation of the dispersion to the solution of stage (1),

(4) substantially removing the auxiliary solvent (G) from the aqueous mixture that is formed, containing auxiliary solvent, with intensive mixing, and

(5) adding a second portion of water to the remaining mixture comprising the blocked polyisocyanates (A) and (B) and the first portion of water, with the provisos that (i) the first portion of water added is of an amount such that the polyisocyanates (A) and (B) form the continuous phase in the mixture which remains after the substantial removal of the auxiliary solvent (G), and (ii) the addition of the second portion of water takes place with intensive mixing, at least up until the point of phase inversion.

Sub B3 6. A stable and high-solids aqueous dispersion containing auxiliary solvent, which comprises:

a disperse phase of a polyisocyanate mixture of from 20-70%

*Sub B3
cont'*

by weight of a blocked, hydrophilically modified polyisocyanate (A) and from 30-80% by weight of a blocked, hydrophobic polyisocyanate (B) having a solids content of from 35-80% by weight.

5 7. The dispersion as claimed in Claim 6, which has a content of auxiliary solvent (G) of less than 25% by weight.

Sub B4

8. A stable and high-solids aqueous dispersion which is virtually free from auxiliary solvent, which comprises:

a disperse phase of a polyisocyanate mixture of from 20-70% by weight of a blocked, hydrophilically modified polyisocyanate (A) and from 30-80% by weight of a blocked, hydrophobic polyisocyanate (B), having a solids content of from 40-60% by weight and a content of auxiliary solvent (G) of <2% by weight.

9. The dispersion as claimed in Claim 8, which has a content of auxiliary solvent (G) of <0.5% by weight.

10. The dispersion as claimed in Claim 6, wherein the blocked, hydrophilically modified polyisocyanate (A) is a blocked, ionic, hydrophilically modified polyisocyanate which forms by complete or partial neutralization of a polyisocyanate 20 having a potentially hydrophilic group.

11. The dispersion as claimed in Claim 10, wherein the potentially hydrophilic group is a carboxyl group and the neutralizing agent (H) is ammonia or an amine.

25 12. The process as claimed in Claim 1, wherein said solvent is acetone, methyl ethyl ketone, tetrahydrofuran, n-butylglycol, di-n-butylglycol or N-methylpyrrolidone.

T05T2E228660
E0
5
10
15
20
25

13. The process as claimed in Claim 10, wherein the polyisocyanate parent of each of polyisocyanates (A) and (B) is a diisocyanate selected from the group consisting of 1,6-diisocyanatohexane (HDI), bis(4-isocyanatocyclohexyl)methane (HMDI), 1,5-diisocyanato-2-methylpentane (MPDI), 1,6-diisocyanato-2,4,4-trimethylhexane and/or 1,6-diisocyanato-2,2,4-trimethylhexane (TMDI), 3-isocyanatomethyl-3,5,5-trimethylcyclohexyl isocyanate (IPDI), uretdione thereof, polyisocyanates having more than two isocyanate groups per molecule prepared by allophanatization, trimerization, biuretization or urethanization of the diisocyanates with polyhydric alcohols, with polyfunctional polyamines, the triisocyanurates prepared by trimerizing the diisocyanates, or aromatic polyisocyanates.

14. The process as claimed in Claim 1, wherein the hydrophilicizing agent is a hydroxycarboxylic, hydroxysulfonic, hydroxyphosphonic, aminocarboxylic or aminosulfonic acids or a tertiary aminoalkanol.

15. The process as claimed in Claim 14, glycolic acid, betaaminopropionic acid, hydroxyethanesulfonic acid, hydroxyethanephosphonic acid, N,N-dimethylaminoethanol or dimethylolpropionic acid (DMPA).

16. The process as claimed in Claim 5, wherein the amount of said first portion of water is not more than 70 wt% of the total of the amount of water added to form the product dispersion.

5

~~17. The process as claimed in Claim 5, wherein at the addition of said first portion of water the temperature is increased to 50-110°C under atmospheric or reduced pressure under intensive mixing which results in the removal of water to a concentration of <2% by weight.~~

18. The process as claimed in Claim 6, wherein solvent is removed to a concentration of <0.5% by weight.

19. A method of preparing a film-forming resin, comprising:
combining the dispersion of Claim 6, as a cross-linking
agent, with an aqueous film-forming resin in which the resin
contains an average of >1.5 NCO-reactive groups in each molecule.

20. The method of Claim 19, wherein said film-forming resin is a melamine resin.

Rodol B5